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IS 9355 (1980): Butachlor, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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IS : 9355 - 1980

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Indian Standard

SPECIFICATION FOR BUTACHLOR, TECHNICAL

(Second Reprint AUGUST 1997)

UDC 632.951 BUTACHLOR

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *November* 1980

Indian Standard

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(*Continued on page* 9)

AMENDMENT NO. 1 JULY 1987
TO
IS : 9355-1980 SPECIFICATION FOR BUTACHLOR, TECHNICAL

(*Page* 4, *Table* 1) — Substitute 'IS : 6940-1982*' *for* 'IS : 6940-1973*', wherever occurs.

(*Page* 4, *Table* 1, *foot-note*) — Substitute the following for the existing foot-note:

'*Method of tests for pesticides and their formulations (*first revision*).'

(*Page* 5, *clause* 4.1 *and Note*) — Substitute the following for the existing matter:

"4.1 Representative samples of the material shall be drawn as per IS : 10946-1984 'Methods of sampling for technical grade pesticides'."

(*Pages* 6 to 8, *Appendix* A) — Substitute the following for the existing appendix:

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF BUTACHLOR CONTENT

A-0. GENERAL

A-0.1 Either of the two methods, namely gas chromatographic method (*see* A-1) or potentiometric titration method (*see* A-2) may be used for determination of butachlor content. However, the GLC method will be the referee method in case of dispute.

A-1. GAS CHROMATOGRAPHIC METHOD

A-1.1 Principle of the Method — The method consists of injecting a sample with an internal standard in a known proportion into a gas chromatograph and determining the area under each peak. The area under the peak is proportional to the mass of the sample. By comparison of this area with that of the standard, the percentage purity of the sample is determined.

A-1.2 Apparatus

A-1.2.1 *Gas Liquid Chromatograph* — Equipped with a recorder and a disc integrator (optional) operating on following suggestive parameters. These parameters can be varied depending upon instrument, provided standardization is done.

Detector	Flame Ionization Detector (FID)	
Column	Stainless steel 2 m × 3 mm i.d. packed with 10 percent DC-200 on chromosorb W-HP, 80-100 mesh	
Temperature	Column	240°C
	Injection	270°C
	Detector	240°C
Carrier gas	Nitrogen	
Flow rate	40 ml/min	

NOTE — Operating parameter as well as attenuation and chart speed shall be adjusted to obtain optimum response and reproducibility.

A-1.2.2 *Micro Syringe* — 2·0 μl-capacity.

A-1.3 Reagents

A-1.3.1 *Standard Reference Butachlor* — of known 99 plus percent purity.

A-1.3.2 *Acetone*

A-1.3.3 *Internal Standard* — bis (2 ethyl-hexyl) adipate.

A-1.4 Procedure

A-1.4.1 *Preparation of Standard Solution* — Weigh accurately 1·0 g of standard reference butachlor into a 100-ml volumetric flask and dilute it with acetone up to the mark.

A-1.4.2 *Preparation of Internal Standard* — Weigh accurately 1·0 g of internal standard adipate into a 100-ml volumetric flask and dilute it with acetone up to the mark.

A-1.4.3 *Preparation of Test Solution* — Weigh accurately the quantity of sample containing about 1·0 g of butachlor into a 100-ml volumetric flask and dilute it with acetone up to the mark.

A-1.4.4 *Analysis of Sample* — Check the chromatograph for thermal and flow equilibrium. Inject alternatively 2-μl of solution prepared by mixing 1 ml of standard solution (**A-1.4.1**) and 1 ml of internal standard (**A-1.4.2**) and 2 μl of solution prepared by mixing 1 ml of test solution (**A-1.4.3**) and 1 ml of internal standard (**A-1.4.2**) at least thrice.

A-1.4.5 Measure the peak heights of areas of butachlor and internal standard solution. Determine the R_f value for each injected standard-internal standard solution.

A-1.5 Calculation

$$\text{Butachlor content, percent by mass} = \frac{M_1 \times A_2 \times 100}{M_2 \times A_1} \times ACF$$

where

M_1 = corrected mass, in g, of the internal standard taken for the test,

A_2 = peak height or area corresponding the butachlor;

M_2 = mass of sample, in g, taken for the test; and

A_1 = peak height or area corresponding to the internal standard.

and

$$ACF = \frac{M_3 \times P_1 \times H_1}{M_4 \times H_2 \times P_2}$$

where

M_3 = mass, in g, of butachlor standard;

P_1 = percent purity of butachlor standard;

H_1 = peak height or area corresponding to the internal standard;

M_4 = mass, in g, of the internal standard taken for the test;

H_2 = peak height or area corresponding to butachlor standard; and

P_2 = percent purity of the internal standard.

A-2. POTENTIOMETRIC TITRATION METHOD

A-2.1 Principle — Refluxing with aniline quantitatively removes the chlorine from butachlor. The ionic chlorine is then titrated against silver nitrate potentiometrically. This gives total chlorine from which butachlor concentration can be computed.

A-2.2 Apparatus

A-2.2.1 *pH Meter* — capable of performing potentiometric titration.

A-2.2.2 *Electrode System* — 0·5 M K_2SO_4, Hg_2SO_4 : Hg.

A-2.3 Reagents

A-2.3.1 *Acetone*

A-2.3.2 *Sulphuric Acid* — 50 percent (*m/m*).

A-2.3.3 *Silver Nitrate* — 0·1 N.

A-2.3.4 *Aniline*

A-2.4 Procedure

A-2.4.1 Weigh accurately about 0·5 g of the sample in a 10 ml pear-shaped flask fitted with a ground-glass joint. Add 3 ml of aniline and a carborundum, boil and reflux vigorously for 30 minutes using ground glass joint water condenser. Cool, add 10 ml acetone through the condenser, extract the contents of the flask in a 250-ml beaker with small portions of acetone and one rinse of 5 to 7 ml of distilled water. Dilute to 100 ml with acetone. Acidify with 7 ml of sulphuric acid. Aniline sulphate will precipitate immediately. Keep the beaker on a magnetic stirrer, add the stirrer bar to the beaker and stir magnetically. Precipitate of aniline sulphate will dissolve. Cool to 15°C and titrate potentiometrically with silver nitrate.

NOTE — The starting potential will be negative 430 to 450 mV and the end point signified by the largest potential dropper increment of nitrate will be in the neighbourhood of 150 mV.

A-2.5 CALCULATION

$$\text{Butachlor content, percent by mass} = \frac{V \times 3{\cdot}118 \times f}{M}$$

where

V = Volume of silver nitrate used in titration;

$f = \dfrac{\text{Percent butachlor (GLC/ basis) in technical material}}{\text{Percent butachlor (chlorine basis) in technical material}}$; and

M = Mass, in g, of the sample taken for the test.

AMENDMENT NO. 2 JULY 1989
TO
IS : 9355 - 1980 SPECIFICATION FOR BUTACHLOR, TECHNICAL

[*Page* 6, *clause* **A-0.1** (*see also* Amendment No. 1)] — Substitute the following for the existing matter:

'**A-0.1** A gas chromatographic method shall be used for determination of butachlor content.'

[*Page* 8, *clause* **A-2** (*see also page* 3, *clause* **A-2** *of* Amendment No. 1)] — Delete 'A-2 POTENTIOMETRIC TITRATION METHOD'.

(AFCDC 6)

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AMENDMENT NO. 3 DECEMBER 1995
TO
IS 9355 : 1980 SPECIFICATION FOR BUTACHLOR, TECHNICAL

(*Page* 4, *clause* 2.1) — Substitute the following for the existing clause:

'**2.1 Description** — The material shall be in the form of a clear, yellow to orange aromatic liquid. 'On storage, the colour of the material may be changed to pink or purple. It shall be free from extraneous matter.'

(*Page* 4, *clause* 3.1) — Substitute 'IS : 8190 (Part 2) - 1988*' *for* 'IS : 8190 (Part II) - 1980*'.

(*Page* 4, *foot-note with* '*' *mark*) — Substitute '(*second revision*)' *for* '(*first revision*)' at the end of text.

(*Page* 5, *clause* 5.2) — Substitute 'IS 1070 : 1992*' *for* 'IS : 1070 - 1977*'

(*Page* 5, *foot-note with* '*' *mark*) — Substitute 'Reagent grade water (*third revision*)' for the existing title.

(FAD 1)

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Indian Standard

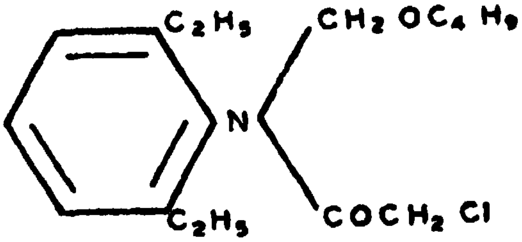
SPECIFICATION FOR BUTACHLOR, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 20 June 1980, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Butachlor is a herbicide used in the formulations meant for agriculture.

0.3 Butachlor is the proposed common name by the International Organization for Standardization (ISO) for 2-chloro-2′, 6′-diethyl-N-butoxymethyl acetanilide. The empirical and the structural formulae and the molecular mass of butachlor are indicated below:

Empirical formula	*Structural formula*	*Molecular Mass*
$C_{17}H_{26}ClNO_2$		311·85

0.4 In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968, and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for butachlor, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a clear, yellow to orange aromatic liquid free from extraneous matter.

2.2 The material shall also comply with the requirements specified in Table 1.

TABLE 1 REQUIREMENTS FOR BUTACHLOR, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST	REF TO
			Appendix of this standard	Cl No. of IS : 6940 - 1973*
(1)	(2)	(3)	(4)	(5)
i)	Butachlor content, percent by mass, *Min*	85	A	—
ii)	Moisture content, percent by mass, *Max*	0·3	—	4
iii)	Acidity (as H_2SO_4), percent by mass, *Max*	0·2	—	11.3.2
iv)	Material insoluble in acetone, percent by mass, *Max*	0·15	—	9

*Methods of tests for pesticides and their formulations.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as per requirements given in IS : 8190 (Part II) - 1980*.

---

*Requirements for packing of pesticides: Part II Liquid pesticides (*first revision*).

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to any other information, as is necessary under the Insecticides Act and Rules:

a) Name of the material;

b) Name of the manufacturer;

c) Date of manufacture;

d) Batch number;

e) Net mass of contents;

f) Butachlor content, percent (*m/m*); and

g) The cautionary notice as worded in Insecticides Act and Rules.

3.2.1 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The representative samples of the material shall be drawn as prescribed in ' Indian Standard Methods for sampling of pesticides and their formulations ' (*under preparation*).

NOTE — Till such time the standard under preparation is published, the samples shall be drawn as agreed between the concerned parties.

5. TESTS

5.1 Tests shall be carried out as prescribed in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070 - 1977*) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Specification for water for general laboratory use (*second revision*).

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF BUTACHLOR CONTENT

A-0. GENERAL

A-0.1 Either of the two methods, namely gas chromatographic method or potentiometric titration method may be used for determination of butachlor content.

A-1. GAS CHROMATOGRAPHIC METHOD

A-1.1 Principle — The butachlor content is determined using an internal standard on a gas chromatograph equipped with a thermal conductivity detector.

A-1.2 Apparatus

A-1.2.1 *Gas Liquid Chromatograph* — equipped with thermal conductivity detector and a suitable recorder with an electronic integrator.

Detector	Thermal Conductivity Detector (TCD)
Column	Stainless steel 1·2 m × 3 mm packed with 10 percent OV-4 on 100-120 mesh chromosorb
Temperature	
Column	220°C
Injection	250°C
Detector	250°C
Carrier gas	Helium
Flow rate	50-60 ml/min

NOTE — Operating parameter as well as attenuation and chart speed shall be adjusted to obtain optimum response and reproducibility.

A-1.2.2 *Micro syringe*—2 μl-capacity.

A-1.3 Reagents

A-1.3.1 *Standard Reference Butachlor* — of known 99 plus percent purity.

A-1.3.2 *Acetone*

A-1.3.3 *Internal Standard* — bis (2 ethyl hexyl adipate).

A-1.4 Procedure

A-1.4.1 *Preparation of the Sample Solution* — Weigh accurately sample equivalent to 0·2 g of butachlor into a small glass stoppered flask or screw cap bottle. Add by pipette internal standard containing 0·2 g bis (2 ethyl hexyl adipate) solution. Close tightly and shake thoroughly to dissolve and extract the butachlor. For coarse or granular materials, shake mechanically for 30 minutes or shake by hand intermittently for 1 hour.

A-1.4.2 *Preparation of Reference Standard* — Weigh accurately 0·2 g of reference butachlor standard into a small glass stoppered flask or screw-cap bottle. Add internal standard solution containing 0·2 g bis (2 ethyl hexyl adipate) and shake to dissolve.

A-1.4.3 *Analysis of the Sample* — Inject 2 µl of the standard and, if necessary, adjust the instrument parameters and the volume injected to give a complete separation within a reasonable time and peak heights from half to three-fourths full scale. Proceed with the determination at least re-injecting each of standard and sample solution in random order.

A-1.4.4 Measure the peak heights or areas of butachlor and internal standard solution. Determine the RF values for each injected standard — internal standard solution.

A-1.5 Calculation

$$\text{Butachlor content, percent by mass} = \frac{M_1 \times P_1 \times A_2 \times 100}{M_3 \times A_1} \times ACF$$

where

M_1 = mass of internal standard taken for the test,

P_1 = percentage purity of the internal standard,

A_2 = peak height or area corresponding to butachlor,

M_3 = mass of sample taken for the test,

A_1 = peak height or area corresponding to the internal standard, and

$$ACF = \frac{M_3 \times A_1 \times P_1}{M_1 \times A_2 \times P_2}$$

where P_2 = percentage purity of butachlor.

A-2. POTENTIOMETRIC TITRATION METHOD

A-2.1 Principle — Refluxing with anilne quantitatively removes the chlorine from butachlor. The ionic chlorine is then titrated against silver nitrate potentiometrically. This gives total chlorine from which butachlor concentration can be computed.

A-2.2 Apparatus

A-2.2.1 *pH Meter* — capable of performing potentiometric titration.

A-2.2.2 *Electrode System* — 0·5 M K_2SO_4, Hg_2SO_4 : Hg

A-2.3 Reagents

A-2.3.1 *Acetone*

A-2.3.2 *Sulphuric Acid* — 50 percent (*m/m*).

A-2.3.3 *Silver Nitrate* — 0·1 N.

A-2.3.4 *Aniline*

A-2.4 Procedure

A-2.4.1 Weigh accurately about 0·5 g of the sample in a 10 ml pear-shaped flask fitted with a ground-glass joint. Add 3 ml of aniline and a carborundum, boil and reflux vigorously for 30 minutes using ground glass joint water condenser. Cool, add 10 ml acetone through the condenser, extract the contents of the flask in a 250-ml beaker with small portions of acetone and one rinse of 5 to 7 ml of distilled water. Dilute to 100 ml with acetone. Acidify with 7 ml of sulphuric acid. Aniline sulphate will precipitate immediately. Keep the beaker on a magnetic stirrer, add the stirrer bar to the beaker and stir magnetically. Precipitate of aniline sulphate will dissolve. Cool to 15°C and titrate potentiometrically with silver nitrate.

NOTE — The starting potential will be negative 430 to 450 mV and the end point signified by the largest potential dropper increment of nitrate will be in the neighbourhood of 150 mV.

A-2.5 CALCULATION

$$\text{Butachlor content, percent by mass} = \frac{V \times 3{\cdot}118 \times f}{M}$$

where

V = Volume of silver nitrate used in titration;

$f = \dfrac{\text{Percent butachlor (GLC/basis) in technical material}}{\text{Percent butachlor (chlorine basis) in technical material}}$; and

M = Mass, in g, of the sample taken for the test.

(*Continued from page* 2)

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